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RECENTLY PUBLISHED RESEARCH OF THE  
LENINGRAD PHARMACEUTICAL RESEARCH INSTITUTE

"Alkalimetric Determination of Sulfapyridine," N. V. Khromov-Borisov, Leningrad Phar Res Inst

"Farmatsiya" Vol 8, No 6, 1945, pp 31-3

Sulfapyridine is hydrolyzed 30 minutes in 25% aqueous HCl by boiling under a reflux. The system is then evaporated to dryness in the same flask on a water bath. The residue (sulfanilic acid and  $\alpha$ -aminopyridine-HCl) is taken up in hot water and titrated with 0.1 N alkali against phenolphthalein to a pink color. This method eliminates the need for titrating in acetone. Accuracy is indicated by five determinations on one sample; the average result was 99.5% (range 99.3-9.7%).

"Colorimetric Determination of Ipecac Root in Small Quantities," Yu. N. Rozemblyum, Leningrad Phar Res Inst

"Farmatsiya" Vol 8. No 2, 1945, pp 21-5

Emetine can be assayed in ipecac root (I) by its reaction with diazotized sulfanilic acid to form an azo dye. The method can be adapted to determination of small amounts of I in dry preparations from other roots. The colorimetric standard is derived from powdered I of known emetine content.

"Gasometric Method for the Determination of Sulfamides,"  
N. V. Khromov-Borisov, Leningrad Phar Res Inst

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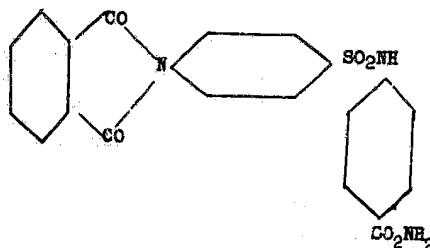
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"Zhur Priklad Khimii" Vol 18, 1945, pp 612-23

Sulfonamides react with  $\text{HNO}_3$  in concentrated  $\text{H}_2\text{SO}_4$  at room temperature to yield  $\text{N}_2\text{O}$  quantitatively; acylated sulfonamides react similarly, the first reaction consists of the cleavage of the acyl group. N-aryl-substituted sulfonamides fail to yield  $\text{N}_2\text{O}$  under these conditions. The accuracy of a gasometric method based on the formation of  $\text{N}_2\text{O}$  was checked on 27 various sulfonamides. The preparation of the following sulfonamides is given as new compounds. Phthalanil (56 g) was added to 150 g  $\text{ClSO}_3\text{H}$  and the mixture was heated to  $70^\circ$  for 1 hour; after pouring into water there was obtained a paste of phthalanilsulfonyl chloride containing 26.4% of the chloride. 134.1 g of the paste, 17.2 g sulfanilamide, 7.8 g  $\text{Na}_2\text{CO}_3$  in 32 cc water, and 170 cc saturated solution of  $\text{NaCl}$  gave  $60-5^\circ$  85% phthaloyldisulfanilamide (I) m  $282-4^\circ$  (from formic acid).



Sulfanilamide (0.7 g) and 0.6 g p-dimethylamino-benzaldehyde were heated to  $210-20^\circ$  for 20 minutes to yield the corresponding  $\text{N}^4$  (p-dimethylamino-benzylidene) sulfanilamide, m  $192-4^\circ$  (from  $\text{EtOH}$ ); the  $\text{HCl}$  salt was obtained by conducting the reaction in aqueous  $\text{HCl}$ , m  $243^\circ$  (with decomposition). p-Chlorobenzenesulfonamide (10 g) heated to  $160^\circ$  with 10 g  $\text{BzCl}$  for 10-15 minutes, then to  $200^\circ$ , gave 14.3 g N-benzoyl-p-chlorobenzenesulfonamide, m  $184-5.5^\circ$  (from 85% formic acid). The product on treatment with  $\text{KNO}_3$  in  $\text{H}_2\text{SO}_4$  gave  $\text{N}_2\text{O}$  and 3-nitro-4-chlorobenzenesulfonic acid K salt, m  $325-6^\circ$  (decomposition, from water).

"Phytosterol as a Ointment Base," A. M. Khaletskiy, L. N. Fedorova, Leningrad Phar Res Inst

"Farmatsiya" Vol 8, No 2, 1945, pp 18-21

A homogeneous ointment base was prepared from 12-15% pine phytosterol and 85-8% water. It did not separate in 30 days at  $20^\circ$ . The paste can be dried and regenerated with warm water. It appears satisfactory as a base for  $\text{ZnO}$ , ichthyol, or mercurial ointments.

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"Determination of Sulfapyridine as the Silver Salt,"  
N. V. Khromov-Borisov, I. M. Yurist, L. P. Popova,  
Leningrad Phar Res Inst

"Farmatsiya" Vol 9, No 1, 1946, pp 26-8

Argentometric determination of sulfapyridine can be carried out gravimetrically or by the Volhard titration. For the former, add about 0.4-g sample to a little less than the equivalent quantity of 0.1 N NaOH and add 50 ml water. Heat to 70° until dissolved, add 25 ml 0.1 N AgNO<sub>3</sub>, cool, filter through fritted glass (No 3), wash free from Ag<sup>+</sup> and then with 300 cc water, and dry at 103°. For the Volhard determination (0.3-g sample), heat again nearly to boiling after precipitating the Ag salt; wash with 5% HNO<sub>3</sub>, then with water until neutral, then with hot water until free from Ag<sup>+</sup> and titrate the excess Hg<sup>+</sup>.

"Preparing Standard Dry Extracts of Thermopsis,"  
A. M. Khaletskii, L. N. Fedorova, Leningrad Phar  
Res Inst

"Farmatsiya" Vol 9, No 5, 1946, pp 32-4

Dry extract of Thermopsis contained 2.85-3.98% alkaloid. For preparation of a standard extract and a nonhygroscopic powder, the Thermopsis was blended with lactose to 0.414% alkaloid (analysis of Thermopsis plants showed 0.42%). This dry extract keeps well in closed vessels, but absorbs moisture if exposed to air. When properly stored, it is stable for 6 months or longer.

"Argentometric Determination of Sulfa Drugs: I. Determination of Sulfidine and Sulfazole," Ya. A. Perel'man, V. I. Kozlova, Leningrad Phar Res Inst

"Farmatsiya" Vol 10, No 1, 1947, pp 22-6

Test titrations of sulfidine and sulfazole with AgNO<sub>3</sub> against excess K<sub>2</sub>CrO<sub>4</sub> were accurate within 1%. In the presence of Cl<sup>-</sup> the titrated solution is acidified with HNO<sub>3</sub>, shaken, filtered, and titrated with AgSCN in the presence of Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·24H<sub>2</sub>O. This titration is accurate enough for routine analysis. (20 references)

"An Investigation of the Phytosterol Obtained by Alkaline Hydrolysis of Pinewood Lignin." A. M. Khaletskiy, N. N. Solomonik, Leningrad Chem-Phar Res Inst

"Zhur Obshch Khimii" Vol 17, 1947, pp 1171-84

Crude phytosterol (I) from pinewood was extracted with ether and the portion solvent in ether (69%) was separated by fractional crystallization from EtOH into a mixture of alcohols of high molecular weight. (II),  $\alpha$  65-75° (30 % of I) and a mixture of

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sterols (III), m 118-33° (38.4% of I). Three optically inactive aliphatic alcohols were isolated by fractional crystallization of II from ether, EtOH, and Me<sub>2</sub>CO: lignoceryl alcohol, m 75-7°, 17% of I, oxidized by CrO<sub>3</sub> in AcOH to lignoceric acid, m 82-3°; C<sub>22</sub>H<sub>44</sub>CH<sub>2</sub>OH, m 70.5-1.5°; acetate, m 51-3°; benzoate, m 45-50°; oxidized to C<sub>22</sub>H<sub>44</sub>CO<sub>2</sub>H, m 75-76.6° (Pb salt, m 115-16°); C<sub>23</sub>H<sub>46</sub>CH<sub>2</sub>OH, m 69-70.2°; acetate, m 53-3.8°; benzoate, m 50-3°; oxidized to C<sub>23</sub>H<sub>46</sub>CO<sub>2</sub>H, m 72.5-3.5° (Pb salt, m 114-15.5°). III was 70%  $\beta$ -sitosterol, m 135-5.5°, [ $\alpha$ ]<sub>D</sub> -36° (in CHCl<sub>3</sub>), isolated and purified via the benzoate. III also contained unidentified sterols and a trace of sitosterol (dihydrositosterol). Stigmasterol and  $\alpha_1$ -,  $\alpha_2$ -,  $\alpha_3$ - and  $\gamma$ -sitosterol were absent. The portion of I insolvent in ether (25%) was largely a mixture of metallic salts of lignoceric and other acids.

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